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RECOVERY AND UTILIZATION OF NATURAL APPLE FLAVORS

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THE PRODUCT

The product is a colorless water solution of the volatile constituents of natural apple flavor concentrated from 100- to 150-fold. Even in this concentrated state, however, the actual proportion of flavoring constituents in the solution does not exceed a few tenths of one percent by weight. The odor is mildly pungent, a characteristic common to concentrated flavoring essences. No perceptible changes occur in the product during storage; apparently it will keep indefinitely.

Blending the flavor concentrate with a good grade of filtered apple juice that has been concentrated by vacuum evaporation results in a full-flavored apple juice concentrate, which when reconstituted with water, is indistinguishable in taste and aroma from fresh apple juice.

THE PROCESS

Basis of Process

In our studies, five significant facts were established:

See Attached Supplement

(1) The flavor of fresh apple juice is not impaired by heating it to 320° F. in 3 seconds.

(2) A 10-percent flash vaporization is sufficient to remove all the volatile flavors; in fact, less than 10 percent may be sufficient for practical purposes.

(3) The freshness of the volatile flavor after it is separated from the juice is not affected by prolonged exposure to the boiling temperature of water, such as occurs in a fractional distillation column.

(4) At atmospheric pressure and 70° F., the volatile flavoring constituents can be recovered in the distillate in concentrations up to 150 times that in apple juice.

To prevent fouling of the flavor recovery equipment the juice should be screened. It may be advantageous not to filter it at this stage, as some of the flavoring elements may be eliminated by too complete clarification.

Superheating and Flash Vaporizing the Juice

See Attached Supplement

To obtain approximately 10-percent vaporization, the juice may be heated to about 320° F. under pressure, then released into an atmospheric-pressure flash chamber. A possible alternate method would be to heat and vaporize the juice simultaneously by a single passage through a continuous evaporator. The essential requirement of the process is that the juice be heated rapidly enough to avoid modifying the fresh flavor. The extreme conditions of time and temperature that can be used safely are not yet known, but it was found that heating the juice to 320° F. in 3 seconds had no effect on the flavor.

When pectin-containing solutions, such as apple juice, are heated by passing through a metallic tube held above a certain critical temperature, presumably different for different solutions, a loosely adherent gelatinous film may be deposited on the tube. This occurred in the superheater in our pilot-plant work and greatly reduced the heat transfer in a few hours. The film was quickly removed by pumping water through the tube at the same rate as that previously used for juice. Means for preventing the formation of this film are under investigation.

In the flash-vaporization system, the evaporation takes place when the pressure on the superheated juice is released. The superheat of the liquid is then transformed into latent heat of vaporization. At 320° F. the pressure required to maintain the juice in the liquid phase

3/8-inch diameter was sufficient to effect a 10-fold increase in concentration of volatile flavoring constituents, that is, to 100 times that of the apple juice.

The vapors from the column pass to a total condenser. The product is withdrawn from the condensate receiver at a rate of 1/100 to 1/150 that of the fresh juice feed rate. The remainder of the condensate is returned through an overflow as reflux to the column. The reflux in the pilot plant studies was returned cold to eliminate the need for a reflux preheater, thus simplifying the operation of the column. A small heating coil or reboiler at the base of the column is used to strip off any volatile flavors from the waste water leaving the bottom of the column.

In the pilot plant studies, the temperature of the condensate was maintained at 70° F. At this temperature the maximum attainable concentration of volatile flavors is about 150 times that in the fresh juice, that is, 1 gallon of product contains the volatile flavor of 150 gallons of juice. Attempts to obtain higher concentrations resulted in losses; the unrecovered flavors escaped through the vent along with the noncondensable gases introduced into the system in the apple juice. Below this concentration the losses that occurred by venting these noncondensable gases (which, of course, leave the system saturated with the volatile flavors) were negligible. In one typical run, the rate at which the noncondensable gases were vented from the system was 2 percent of the apple juice-feed rate expressed on a volume basis.

EQUIPMENT REQUIRED

This process for the production of apple flavor would seem suitable for immediate adoption by present manufacturers of bland apple sirups and concentrates. Estimates of equipment requirements and processing costs have been made on that basis. It is assumed that steam at 120 lbs. per sq. in. (gauge) is available for the superheater. If it is not, then instead of installing a new boiler to obtain the 120 lbs. per sq. in., a single-pass, continuous, high-speed evaporator should be considered, because in it the heating and evaporation both take place at atmospheric pressure and low-pressure steam can be used for heating. The cost of the steam for both the superheater and the continuous evaporator, except for the small amount required by the reboiler, can be considered as chargeable to the manufacture of the sirup produced from the juice, because the concentration effected by this preliminary evaporation lessens the load on the sirup evaporators.

The following equipment is required:

Metering pumps	2
Juice superheaters (one a spare)	2
Vapor-liquid separator	1
Fractionating column	1
Condenser	1
Miscellaneous piping and fittings	

Copper is a suitable material for apparatus for manufacturing apple sirup if the surface is kept bright, that is, not allowed to become oxidized by intermittent use. It should also be satisfactory for all the apparatus required for recovery of flavor except the superheater and the metering pumps. The pumps can be bronze, but for the superheater stainless steel is recommended. Apple juice at the high temperature which it reaches in the superheater may slightly attack copper and bronze and thus be contaminated.

The metering pump on the feed line should preferably be duplex or double-acting to reduce the pulsations inherent in reciprocating pumps and even then a small surge chamber may be desirable. The small metering pump on the product line may be a single-acting pump, for the pulsations caused by it are of no consequence. This pump delivers against a low pressure, whereas the large pump on the feed line must be capable of delivering the juice at a pressure sufficient to maintain it in liquid phase up to the flashing orifice as well as to overcome the drop in pressure resulting from the flow of juice through the superheater.

On the basis of present pilot plant data, a single-tube superheater with a tube of 0.334-inch inside diameter (1/2 inch outside diameter, #14 gauge wall) and 38 feet long and with steam at 100 to 120 lbs. per sq. in. (gauge) as the heating medium should superheat 208 gallons of apple juice per hour (5,000 gallons per 24-hour day) to 320° F. in 3 seconds. The pressure drop through such a superheater would not exceed 40 lbs. per sq. in. To maintain the juice in liquid phase, the pressure drop across the flashing orifice must be at least 75 lbs. per sq. in. With proper design this crifice pressure drop will not exceed 160 lbs. per sq. in.; hence, the overall drop in pressure on the juice line from the pump to the low-pressure side of the flashing orifice will not exceed 200 lbs. per sq. in. It is recommended that two superheaters be installed in parallel, each of diameter of a packed column is increased in order to allow for non-uniformity of flow of reflux down the column. Provision must be made for maintaining a small reboiler (coiled, steam-heated tube) immersed in liquid at the base. The amount of reboiling required to prevent loss of flavor in the bottoms product from the column could be reduced by the addition of a length of packed column below the vapor-feed inlet, which would function as a tripping section. It is doubtful, however, whether the saving in heat thus obtained would be significant. The column should be lagged to prevent excessive loss of heat from it.

The condenser should be designed to condense the vapors and then cool the condensate to 70° F. ± 10° F. If cooling water is not available at a low enough temperature, a refrigerated cooler could be placed in series with the condenser to achieve the last few degrees of cooling. In this case better heat economy would be obtained by returning the reflux at whatever temperature it comes from the condenser and using the refrigerated cooler to cool only that portion of the condensate withdrawn as product. For such an arrangement, the vent for non-condensable gases should be located after the liquid cooler but before the product pump.

The product at 70° F. is nearly saturated with volatile flavors. It should therefore be discharged into cooled receivers to avoid loss. After the receiver is filled and tightly capped, it can be stored at room temperature. It would seem to be a desirable precaution, however, to cool the product before it is used.

The cost of producing volatile apple flavor concentrated one hundred times (that is, 1 gallon of such product per 100 gallons of juice certain uses, a slightly modified apple flavor may be preferable. A "baked apple" flavor is easily obtained by prolonging the heating of the juice prior to flavor recovery.

Since much of the apple juice evaporated commercially goes into products where natural apple flavor is of no value, the volatile flavoring constituents now discarded in the preparation of these products could by this new process be obtained in concentrated form and sold as natural flavoring essence. The principles of this new process should also be applicable to the recovery of natural flavors from berries and other fruits.

per sq. in. of steam was used in the steam chest. Under these conditions the juice could be heated for a total time of at least 15 seconds without modification of the fresh flavor, and the rate of processing the juice was 50 gallons per hour. The modification of flavor resulting from doubling this heating tim would not be very significant. However, in general it is desirable to heat th juice for as short a time as possible, consistent with practical evaporator design.

Under certain conditions, as for example when the maximum steam pressure available is much less than 30 lbs. per sq. in., it may be desirable to employ a high velocity preheater. However, when such a preheater with a tube of 0.18 inch inside diameter was used ahead of the evaporator described above, the total permissible heating time without modification of fresh flavor was only 10 seconds when the time for preheating the juice to its boiling point was 2.0 seconds.

With the evaportor system, either alone or with a preheater, fouling is so slight that daily cleaning would probably be sufficient. Moreover, we hav been able to remove in a few minutes the fouling deposit formed in a 3-hour r on the tubes of both the preheater and the evaporator. This is accomplished shutting off the steam, pumping cold water through the heating tubes, then stopping the pumps and suddenly admitting steam to the steam chest. The boiling, that takes place inside the tubes shortly thereafter, removes the deposi